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N-(4-Methoxybenzoyl)-*N′*-*o*-tolylthiourea

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.110
Data-to-parameter ratio = 15.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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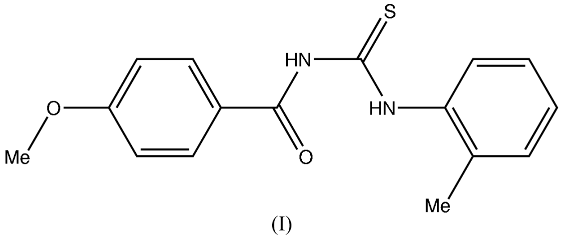
In the title compound, $C_{16}H_{16}N_2O_2S$, the *p*-methoxybenzoyl and *o*-methylphenyl fragments are *trans* and *cis*, respectively, with respect to the thione group across the C—N bonds. The central carbonylthiourea (N_2C_2SO) moiety makes dihedral angles with the *p*-methoxybenzoyl and *o*-methylphenyl fragments of 16.58 (7) and 22.76 (8)°, respectively.

Comment

Recent interest in the study of thiourea derivatives has been driven by their potential as biologically active compounds (Baruah *et al.*, 2002) and as useful agrochemical intermediates (Ramadas *et al.*, 1993). The title compound, (I), is analogous to *N*-benzoyl-*N′*-phenylthiourea (Yamin & Yusof, 2003), but having methoxy and methyl substituents at the *para* and *ortho* positions of the benzoyl and phenyl groups, respectively (Fig. 1).



(I)

The C8—S1, C8—N1 and C8—N2 bond lengths of 1.6608 (15), 1.3925 (19) and 1.3365 (18) Å, respectively, are comparable with those of *N*-benzoyl–N’-phenylthiourea [C—S = 1.6567 (15) Å, C8—N1 = 1.383 (2) Å and C8—N2 = 1.326 (2) Å] and other benzoylthiourea derivatives (Yusof & Yamin, 2004). The *p*-methoxybenzoyl and *o*-methylphenyl groups maintain their *trans* and *cis* positions, respectively, about the thione group across the C8—N1 and C8—N2 bonds, respectively. Other bond lengths and angles are within normal

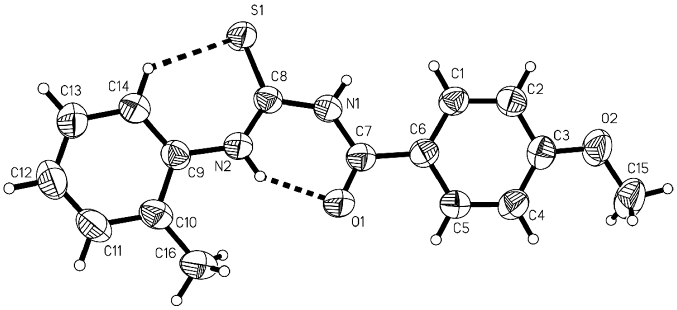


Figure 1
The molecular structure of (I), drawn with 50% probability displacement ellipsoids. Dashed lines indicate intramolecular H bonds.

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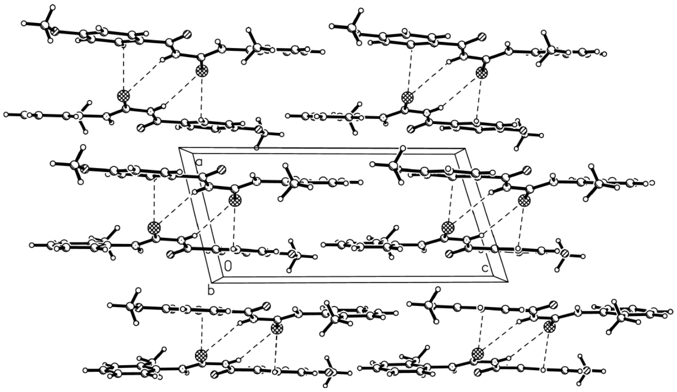

Figure 2
Packing diagram of compound (I). The dashed lines denote N—H···S and C—H···S hydrogen bonds.

ranges (Allen *et al.*, 1987). The central carbonylthiourea (C7/N1/C8/S1/N2/O2), the *p*-methoxybenzoyl (C1–C6/O2/C15) and the *o*-methylphenyl (C9–C14) groups are each planar. The maximum deviation is 0.021 (1) Å for atom N1 from the mean plane of the carbonylthiourea group. The dihedral angles between the central carbonylthiourea moiety and the *p*-methoxybenzoyl and *o*-methylphenyl fragments are 16.58 (7) and 22.76 (8)°, respectively. The inclination between the aryl fragments is 7.58 (9)°.

There are two intramolecular interactions, N2—H2···O1 and C14—H14···S1, present in the molecule. As a result, two pseudo-six-membered rings are formed. In the crystal structure, the molecule is stabilized by weak intermolecular interactions, N1—H1···S1^{i} and C1—H1*A*···S1^{i} [symmetry code: (i) 1 − *x*, 2 − *y*, 2 − *z*; Table 2], forming dimers (Fig. 2).

Experimental

A solution of *o*-methylphenylamine (1.24 g, 0.012 mol) in acetone (50 ml) was added dropwise to an acetone solution (50 ml) containing an equimolar amount of *p*-methoxybenzoylisothiocyanate in a two-necked round-bottomed flask. The solution was refluxed for about 2 h and then cooled in ice. The white precipitate was filtered off and washed with an ethanol–distilled water mixture, then dried in a vacuum (yield 83%). Recrystallization from ethanol yielded single crystals suitable for X-ray analysis.

Crystal data

$C_{16}H_{16}N_2O_2S$
M_r = 300.37
Triclinic, $P\bar{1}$
a = 7.4923 (14) Å
b = 7.7866 (14) Å
c = 14.406 (3) Å
α = 94.492 (3)°
β = 102.639 (3)°
γ = 113.064 (3)°
V = 741.8 (2) Å^3

Z = 2
D_x = 1.345 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 893 reflections
θ = 2.9–26.0°
μ = 0.22 mm^{-1}
T = 273 (2) K
Block, colourless
0.51 × 0.50 × 0.49 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
7569 measured reflections
2896 independent reflections

2681 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.013
$\theta_{\rm max}$ = 26.0°
h = −9 → 9
k = −9 → 9
l = −17 → 17

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.110
S = 1.06
2896 reflections
193 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0615P)^2 + 0.1424P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max}$ = 0.17 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.21 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.023 (6)

Table 1
Selected geometric parameters (Å, °).

S1—C8	1.6608 (15)	N1—C8	1.3925 (19)
O1—C7	1.2282 (18)	N2—C8	1.3365 (18)
N1—C7	1.3767 (18)	N2—C9	1.4170 (19)
N2—C8—N1	114.51 (12)	N1—C8—S1	117.89 (10)
N2—C8—S1	127.60 (11)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14···S1	0.93	2.59	3.1895 (18)	123
N2—H2···O1	0.86	1.87	2.6063 (17)	142
N1—H1···S1^{i}	0.86	2.85	3.6596 (15)	158
C1—H1*A*···S1^{i}	0.93	2.78	3.3712 (17)	122

Symmetry code: (i) 1 − *x*, 2 − *y*, 2 − *z*.

After their location in a difference map, all H atoms were fixed geometrically at ideal positions and allowed to ride on the parent C atoms, with C—H = 0.93–0.96 Å and N—H = 0.86 Å, and with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C) for CH_3, 1.2$U_{\rm eq}$(C) for CH and 1.2$U_{\rm eq}$(N) for NH.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT* and *SADABS* (Sheldrick, 1996); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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